
**Appendix A -
EPA Data Quality Indicators (DQI) Tables for VOCs, SVOCs, Metals, PCBs, PAHs,
Hexavalent Chromium and Mercury**

HEXAVALENT CHROMIUM in Soil and Water
Colorimetric
SW-846 Method 7196A *

HEXAVALENT CHROMIUM in Water
Atomic Absorption, Chelation-Extraction
EPA Method 218.4

Table 1. Summary of Contract Required Detection Limits, Holding Times, and Preservation for Hexavalent Chromium (Cr⁶⁺)

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times ^a	Preservation
Hexavalent Chromium (Cr ⁶⁺) in Water Samples	20 µg/L ^b 10 µg/L ^c	Technical: 24 hours from collection; Contract: 12 hours from receipt at laboratory	Cool to 4°C ±2°C ^e
Cr ⁶⁺ in Soil Samples ^d	2 mg/kg	Contract: 12 hours from receipt at laboratory	Cool to 4°C ±2°C

* If the colorimetric method is not suitable, EPA Method 218.4 may be used. If EPA Method 218.4 is designated for analysis, the diphenylcarbazide colorimetric procedure discussed in Section 2.3 of EPA Method 218.4 **may NOT** be used.

^a Laboratories bidding on this analysis must be located within a 3 hour drive from the site of sample collection.

^b 20 µg/L CRDL for analysis by SW-846 Method 7196A

^c 10 µg/L CRDL for analysis by EPA Method 218.4

^d Soil Samples: Follow EPA SW-846 Method 3060A (January 1995) for sample digestion.

^e Determine initial pH of water samples.

Data Calculations and Reporting Units:

Calculate the sample results from the standard curve. Calculate soil sample results using the equation provided in Section 7.10.1 of Method 3060A. Report water sample results in concentration units of micrograms per liter (µg/L). Report soil sample results in concentration units of milligrams per kilogram (mg/kg). Cr⁶⁺ concentrations that are less than 10 µg/L or 10 mg/kg to 1 significant figure, and Cr⁶⁺ concentrations that are greater than or equal to 10 µg/L or 10 mg/kg to 2 significant figures.

For rounding results, adhere to the following rules:

a) If the number following those to be retained is less than 5, round down;

b) If the number following those to be retained is greater than 5, round up;

or

c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 2. Summary of Calibration Procedures for Hexavalent Chromium

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b}	Initially; whenever required, due to failure of ICV or CCV	$r \leq 0.995$	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Separate source from ICAL standards)	Daily, prior to sample analysis; immediately following ICAL	$\pm 10\%$ from expected concentration	1. Identify and document problem 2. Reprep ICV and reanalyze all associated samples 3. Recalibrate and reanalyze reprep ICV and all associated samples
Continuing Calibration Verification (CCV)	Following ICV and before sample analysis; after every 10 samples and end of run	$\pm 10\%$ from expected concentration	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples with results less than 10 times the level of contamination in the blank.
CRDL Verification Standard	After initial CCV/CCB	$\pm 20\%$ from expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify

^a The ICAL standards for SW-846 Method 7196A must include standard concentrations at the CRDL (20 µg/L) through 500 µg/L.

The ICAL standards for EPA Method 218.4 must include standard concentrations at the CRDL (10 µg/L) through 250 µg/L.

^b A calibration curve must be prepared with each set of samples.

Table 3. Summary of Internal Quality Control Procedures for Hexavalent Chromium

QC Element	Frequency	Acceptance Criteria	Corrective Action
Laboratory Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike (MS)	One per batch or SDG (1 per 20 samples minimum)	± 25% from expected value	1. Flag associated data with an "N"
Laboratory Control Sample (LCS)	One per batch or SDG (1 per 20 samples minimum)	± 20% from expected concentration	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

ORGANOCHLORINE PESTICIDES AND POLYCHLORINATED BIPHENYLS (PCBs)

SW-846 Method 8081 or 8080

Table 1A. Summary of Holding Times and Preservation for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs)

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Water Samples	<u>Technical for Extraction:</u> 7 days from collection; <u>Contract for Extraction:</u> 5 days from receipt at laboratory <u>Technical and Contract for Analysis:</u> 40 days from extraction	Cool to 4EC ±2EC
Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) in Soil Samples	<u>Technical for Extraction:</u> 14 days from collection; <u>Contract for Extraction:</u> 10 days from receipt at laboratory <u>Technical and Contract for Analysis:</u> 40 days from extraction	Cool to 4EC ±2EC

^a Individual target compounds are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the calibration factors (CF) of single component pesticides according to Section 7.4.2 of SW-846 Method 8000A. Calculate sample results using the analyte CFs from the midpoint standard of the associated initial calibration curve. Perform sample quantitation for multiple components pesticides according to Section 7.6 of SW-846 Method 8080A or 8081.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg).

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits (CRQL) for SW-846 Method 8081 or Method 8080

COMPOUND	CAS No.	CRQL Water µg/L	CRQL Soil µg/kg
alpha-BHC	319-84-6	0.05	2
beta-BHC	319-85-7	0.05	2
delta-BHC	319-86-8	0.05	2
gamma-BHC (Lindane)	58-89-9	0.05	2
Heptachlor	76-44-8	0.05	2
Aldrin	309-00-2	0.05	2
Heptachlor epoxide	1024-57-3	0.05	2
Endosulfan I	959-98-8	0.05	2
Dieldrin	60-57-1	0.1	3
4,4'-DDE	72-55-9	0.1	3
Endrin	72-20-8	0.1	3
Endosulfan II	33213-65-9	0.1	3
4,4'-DDD	72-54-8	0.1	3
Endosulfan sulfate	1031-07-8	0.1	3
4,4'-DDT	50-29-3	0.1	3
Methoxychlor	72-43-5	0.5	17
Endrin ketone	53494-70-5	0.1	3
Endrin aldehyde	7421-93-4	0.1	3
alpha-Chlordane	5103-71-9	0.05	2
gamma-Chlordane	5103-74-2	0.05	2
Toxaphene	8001-35-2	5	170
Aroclor-1016	12674-11-2	1	33
Aroclor-1221	11104-28-2	2	67
Aroclor-1232	11141-16-5	1	33
Aroclor-1242	53469-21-9	1	33
Aroclor-1248	12672-29-6	1	33
Aroclor-1254	11097-69-1	1	33
Aroclor-1260	11096-82-5	1	3

Table 2. Summary of Calibration Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20% (#30% for Surrogate compounds)	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	%D between CF of CCV and avg CFs from ICAL #25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Endrin and 4,4'-DDT Breakdown	Beginning and end of analytical sequence	#20% each or #30% combined	1. Investigate source of the problem and document 2. If either Endrin, 4,4'-DDT, or their breakdown products were detected, re-analyze the samples

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b ICAL Prepare initial calibration individual standard mixtures A and B (IND A and IND B) containing the single component pesticides specified in Table 9 of SW-846 Method 8081 at three concentration levels. For multiple response pesticides, including toxaphene and Aroclors (except 1016 and 1260), prepare separate initial calibration standards at the following concentration levels: Aroclors (except 1221) at 100 ng/mL; Aroclor-1221 at 200 ng/mL; and toxaphene at 500 ng/mL. Aroclor-1016 and Aroclor-1260 may be combined into a single standard solution. Spike all calibration standards with the surrogate compounds discussed in Table 3 at a concentration of 20 ng/mL.

^c Report the retention time window for each analyte. For multiple component pesticides, calculate the retention time window for 5 major peaks from the initial calibration standard analysis.

Determine retention time windows for both single and multiple component pesticides using the following guidelines:

<u>Column Type</u>	<u>Retention Time Window in Minutes</u>
Packed Column	#± 2%
Mega bore or wide bore capillary column	±0.05 for tetrachloro-m-xylene through Aldrin ±0.07 for compounds which elute after Aldrin ±0.1 for decachlorobiphenyl

Table 3. Summary of Internal Quality Control Procedures for Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081 or 8080

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRQL for each compound	1. Investigate source of contamination and document 2. Re-extract and re-analyze all samples processed with a non-compliant method blank
Surrogate ^b	Every standard, sample, method blank and QC sample at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD) ^c	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	50-135% of expected value; #30 RPD between MS and MSD	1. Address in narrative

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Spike each standard, sample, and blank with 1mL of a solution containing 0.2 Fg/mL each of tetrachloro-m-xylene and decachlorobiphenyl

^c Spike MS/MSD samples with 1mL of a solution containing the following compounds and levels:

Target compound	Concentration (Fg/mL)	Target Compound Concentration (Fg/mL)
?-BHC	0.5	Heptachlor 0.5
4,4'-DDT	1.0	Aldrin 0.5
Endrin	1.0	Dieldrin 1.0

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Perform confirmation analyses on a column of a phase different from that used for quantitation. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Table 2, above.

POLYNUCLEAR AROMATIC HYDROCARBONS (PAHs)

SW-846 Method 8310

Table 1A. Summary of Holding Times and Preservation for Carbamate and Urea Pesticides by High Performance Liquid Chromatography

Analytical Parameter ¹	Technical and Contract Holding Times	Preservation
Polynuclear Aromatic Hydrocarbons (PAHs)	<u>Technical for Extraction: 7 days</u> from collection; <u>Contract for Extraction: 5 days</u> from receipt at laboratory <u>Technical and Contract for Analysis: 40 days</u> from extraction	Cool to 4EC ±2EC; Store in TFE-fluorocarbon-sealed bottles away from the light
Polynuclear Aromatic Hydrocarbons (PAHs)	<u>Technical for Extraction: 14 days</u> from collection; <u>Contract for Extraction: 10 days</u> from receipt at laboratory <u>Technical and Contract for Analysis: 40 days</u> from extraction	Cool to 4EC ±2EC; Store away from the light

¹ Individual target compounds are listed in Table 1B.

Data Calculations and Reporting Units:

Calculate the sample results using calibration factors determined according to Sections 7.4.2 and 7.8.1 of SW-846 Method 8000A.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg).

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 1B. Target Compound List, CAS Numbers, and Contract Required Quantiation Limits for SW-846 Method 8310

COMPOUND	CAS No.	CRQL Water µg/L	CRQL Soil µg/kg
Acenaphthene	83-32-9	2	1340
Acenaphthylene	208-96-8	2	1340
Anthracene	120-12-7	0.1	67
Benzo(a)anthracene	56-55-3	0.1	67
Benzo(a)pyrene	50-32-8	0.1	67
Benzo(b)fluoranthene	205-99-2	0.1	67
Benzo(g,h,i)perylene	191-24-2	0.1	67
Benzo(k)fluoranthene	207-08-9	0.1	67
Chrysene	218-01-9	0.1	67
Dibenzo(a,h)anthracene	53-70-3	0.1	67
Fluoranthene	206-44-0	0.1	67
Fluorene	86-73-7	2	1340
Indeno(1,2,3-cd)pyrene	193-39-5	0.1	67
Naphthalene	91-20-3	2	1340
Phenanthrene	85-01-8	0.1	67
Pyrene	129-00-0	0.1	67

Table 2. Summary of Calibration Procedures for Polynuclear Aromatic Hydrocarbons by SW-846 Method 8310

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20%	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	%D between CF of CCV and avg CFs from ICAL #15%	1. Re-calibrate and verify 2. Re-analyze samples back to last compliant CCV
Retention time evaluation for CCV standards	Each analysis of CCV standards	± 3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last compliant CCV

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.

^c Report the retention time window for each analyte. Determine retention time windows as ± 3 x the standard deviation of the average initial calibration retention time for each analyte.

Table 3. Summary of Internal Quality Control Procedures for Polynuclear Aromatic Hydrocarbons by SW-846 Method 8310

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRQL for each compound	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-extracted and re-analyzed
Surrogate ^b	Every standard, sample, and method blank at 10 times CRQL	65-125% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Duplicate (MS/MSD) ^c	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	75-125% of expected value; #30 RPD between MS and MSD	1. Report in case narrative
QC Check Solution	One per Batch or SDG	See Table 3 of SW-846 Method 8310	1. Repeat preparation and analysis of QC check solution.
Cleanup Standard (midpoint concentration)	When column cleanup is used	>85% Recovery	1. Investigate problem, determine cause, and document. 2. Do not analyze samples until cleanup standard is compliant.

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b The compound decafluorobiphenyl is recommended.

^c MS/MSD spike should contain a minimum of three PAH compounds chosen from the compound list in Table 1B.

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

pH in Liquid and Soil
SW-846 Method 9040 (Liquid) and SW-846 Method 9045 (Soil)

Table 1. Summary of Holding Times and Preservation for liquid and soil pH

Analytical Parameter	Technical and Contract Holding Times	Preservation
pH (Liquid and Soil)	Analyze pH immediately upon sample receipt by the laboratory	Cool to 4EC ±2EC

For soil samples:

In addition to the procedure outlined in Section 7.2 of SW-846 method 9045C,

For noncalcareous soils:

1. Weigh 20 grams of soil in a 50 mL beaker. Add 20 mL of Type II water. Stir the resulting suspension several times during the next 30 minutes.
2. Let the suspension to stand undisturbed for about 1 hour to allow most of the suspended clay to settle out

For calcareous soils:

1. Weigh 10 grams of soil in a 50 mL beaker. Add 20 mL of 0.01M CaCl₂. Stir the resulting suspension several times during the next 30 minutes.
2. Let the suspension to stand undisturbed for about 1 hour to allow most of the suspended clay to settle out.

Data Calculations and Reporting Units:

Read pH meter results directly in pH units and solution temperature in EC.

Report pH results to the nearest 0.1 pH unit and temperature to the nearest EC. Report the results for noncalcareous soils as "soil pH measured in water" and for calcareous soils, report the results as "soil pH measured in 0.01M CaCl₂".

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis must be legible and sufficient to validate all sample and QC analyses.

Table 2. Summary of Calibration Procedures for pH by SW-846 Method 9045 (Soil) and SW-846 Method 9040 (Liquids) and

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Meter Standardization	Initially	Refer to manufacture's instructions	Refer to manufacture's instructions concerning standardization and electrode(s) care
Calibration	Daily, prior to sample analysis	A minimum of 2 primary or secondary standard buffers must be used; Buffers must bracket expected pH of sample; Buffers must be 3 or more pH units apart; Buffer readings must be within 0.05 pH units of buffer's true value	<ol style="list-style-type: none"> 1. Allow buffer temperatures to reach equilibrium 2. Repeat measurement with successive volumes of fresh buffer until acceptance criteria are met 3. Replace electrode(s) (follow manufacture's instructions)
Buffer pH Check	Check a calibration buffer solution after every 10 samples (minimum)	<±0.1 pH unit difference	<ol style="list-style-type: none"> 1. Repeat measurement with successive volumes of fresh buffer until acceptance criteria is met before continuing analysis 2. Recalibrate meter and reanalyze samples

NOTE: Electrode(s) must be cleaned after each sample analysis by thoroughly rinsing and gently wiping.

Table 3. Summary of Internal Quality Control Procedures for pH by SW-846 Method 9040 (Liquids) and SW-846 Method 9045 (Soil)

QC Element	Frequency	Acceptance Criteria	Corrective Action
Sample Temperatures	Every sample	<2 EC difference from buffer	Refer to manufacture's instructions concerning the pH meter temperature compensating function
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum) ^a	<±0.1 pH unit difference	<ol style="list-style-type: none"> 1. Allow sample temperatures to reach equilibrium 2. Perform buffer pH check (See Calibration Procedure) 3. Clean electrode (See Calibration Procedure Note)

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

SEMIVOLATILE ORGANIC COMPOUNDS (SVOCs)

SW-846 Method 8270

Table 1A. Summary of Holding Times and Preservation for Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Semivolatile Organic Compounds (SVOCs) in Water	<u>Technical for Extraction:</u> 14 days from collection; <u>Contract for Extraction:</u> 10 days from receipt at laboratory; <u>Technical and Contract for Analysis:</u> 40 days from extraction	Cool to 4EC ±2EC;
SVOCs in Soil ^b	<u>Technical for Extraction:</u> 14 days from collection; <u>Contract for Extraction:</u> 10 days from receipt at laboratory;	Cool to 4EC ±2EC
	<u>Technical and Contract for Analysis:</u> 40 days from extraction	

^a Individual target compounds are listed in Table 1B.

^b Perform initial sample analysis using a 2-gram sample for mid-level analysis and a 30-gram sample for low-level analysis

Data Calculations and Reporting Units:

Use the mean RRF from the initial calibration to calculate the concentration of individual analytes according to Section 7.7.2 of EPA Method 8270C, Revision 3.0.

Report water sample results in concentration units of micrograms per liter (Fg/L). Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B: Target Compound List and Contract Required Quantitation Limits (CRQLs) for Semivolatile Organic Compounds (SVOCs) by SW-846 Method 8270

Analyte	Contract Required Quantitation Limits		PRGs	
	Water (Fg/L)	Soil (mg/kg)	Industrial Soil (mg/kg)	Tap Water (Fg/L)
1,2-Dichlorobenzene	10	0.33	370	370
1,2,4-Trichlorobenzene	10	0.33	1,700	190
1,3-Dichlorobenzene	10	0.33	140	180
1,4-Dichlorobenzene	10	0.33	7.3	0.47
2-Chloronaphthalene	10	0.33	NA	NA
2-Chlorophenol	10	0.33	240	38
2-Methylnaphthalene	10	0.33	NA	NA
2-Methylphenol	10	0.33	53,000	1,800
2-Nitroaniline	25	0.8	64	2.2
2-Nitrophenol	10	0.33	NA	NA
2,2'-oxybis (1-Chloropropane)	10	0.33	NA	NA
2,4-Dichlorophenol	10	0.33	3,200	110
2,4-Dimethylphenol	10	0.33	21,000	730
2,4-Dinitrophenol	25	0.8	2,100	73
2,4-Dinitrotoluene	10	0.33	2,100	73
2,4,5-Trichlorophenol	25	0.8	110,000	3,700
2,4,6-Trichlorophenol	10	0.33	270	6.1
2,6-Dinitrotoluene	10	0.33	1,100	37
3-Nitroaniline	25	0.8	NA	NA
3,3'-Dichlorobenzidine	10	0.33	6.7	0.15
4-Bromophenyl-phenylether	10	0.33	NA	NA
4-Chloro-3-methylphenol	10	0.33	NA	NA
4-Chloroaniline	10	0.33	4,300	150
4-Chlorophenyl-phenyl ether	10	0.33	NA	NA
4-Methylphenol	10	0.33	5,300	180
4-Nitroaniline	25	0.8	NA	NA
4-Nitrophenol	25	0.8	66,000	2,300
4,6-Dinitro-2-methylphenol	25	0.8	NA	NA
Acenaphthene	10	0.33	28,000	370
Acenaphthylene	10	0.33	NA	NA
Anthracene	10	0.33	220,000	1,800
Benzo (a) anthracene	10	0.33	3.6	0.092
Benzo (a) pyrene	10	0.33	0.36	0.0092
Benzo (b) fluoranthene	10	0.33	3.6	0.092
Benzo (g, h, i) perylene	10	0.33	NA	NA
Benzo (k) fluoranthene	10	0.33	36	0.92
bis (2-Chloroethoxy) -methane	10	0.33	NA	NA

Table 1B: Target Compound List and Contract Required Quantitation Limits (CRQLs) for Semivolatile Organic Compounds (SVOCs) by SW-846 Method 8270

Analyte	Contract Required Quantitation Limits		PRGs	
	Water (Fg/L)	Soil (mg/kg)	Industrial Soil (mg/Kg)	Tap Water (Fg/L)
bis(2-Chloroethyl) ether	10	0.33	0.56	0.0098
bis(2-Ethylhexyl)phthalate	10	0.33	21	4.8
Butylbenzylphthalate	10	0.33	930	7,300
Carbazole	10	0.33	150	3.4
Chrysene	10	0.33	360	9.2
Di-n-butylphthalate	10	0.33	NA	NA
Di-n-octylphthalate	10	0.33	10,000	730
Dibenz(a,h)anthracene	10	0.33	0.36	0.0092
Dibenzofuran	10	0.33	3,200	24
Diethylphthalate	10	0.33	100,000	29,000
Dimethylphthalate	10	0.33	100,000	370,000
Fluoranthene	10	0.33	37,000	1,500
Fluorene	10	0.33	22,000	240
Hexachlorobenzene	10	0.33	1.9	0.042
Hexachlorobutadiene	10	0.33	38	0.86
Hexachlorocyclopentadiene	10	0.33	7,100	260
Hexachloroethane	10	0.33	210	4.8
Indeno(1,2,3-cd)pyrene	10	0.33	3.6	0.092
Isophorone	10	0.33	3,200	71
N-Nitroso-di-n-propylamine	10	0.33	0.43	0.0096
N-nitrosodiphenylamine	10	0.33	610	14
Naphthalene	10	0.33	190	6.2
Nitrobenzene	10	0.33	100	3.4
Pentachlorophenol	25	0.8	15	0.56
Phenanthrene	10	0.33	NA	NA
Phenol	10	0.33	100,000	22,000
Pyrene	10	0.33	26,000	180

Notes:

- Fg/L = micrograms per liter.
- mg/kg = milligrams per kilogram.
- NA = Not available.
- PRG = U.S. EPA Preliminary Remediation Goals, Region 9 May 1, 1998..

Table 2. Summary of Calibration Procedures for SVOCs by SW-846 Method 8270C

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with decafluorotriphenylphosphine (DFPPP)	Beginning of each 12-hour period during which standards samples are analyzed	Ion abundance criteria in Table 3 of Method 8270C, Revision 3.0	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for RRFs #30%; or correlation coefficient (r) generated by the linear regression must be 0.99 for all analytes	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV) ^d	Beginning of every 12-hour period, and end of run	%D between RRF of CCV and avg RRFs from ICAL #30%; or $\pm 30\%$ of true value for linear regression	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Integrated areas of Internal Standards	Each analysis	Area must be within -50 to 100 percent.	1. Re-analyze samples with internal standard -50 percent and greater than 100 percent
Retention time evaluation of all standard, surrogate, and sample analytes	Each analysis	$\pm 3 \times$ the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples out control limits

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.

^c Report the retention time window for each analyte. Determine retention time windows as $\pm 3 \times$ the standard deviation of the average initial calibration retention time for each analyte.

^d If some compounds are beyond the control limits of the CCV and these target compounds are detected in samples and 10 percent or less of these analytes are beyond control limits, a single point calibration may be used to quantify the out-of-control analytes.

Table 3. Summary of Internal Quality Control Procedures for SVOCs by SW-846 Method 8270C

QC Element	Frequency	Acceptance Criteria ^b	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG ^a	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Re-analyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	<u>Water Sample:</u> MS and MSD recoveries and RPD between within laboratory limits <u>Soil Sample:</u> MS and MSD recoveries and RPD between within laboratory limits	1. Report in case narrative
Surrogate Spikes	Every sample, standard and method blank	<u>Water Sample:</u> Surrogate recoveries within laboratory limits <u>Soil Sample:</u> Surrogate recoveries within laboratory limits	1. Re-analyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	<u>Water Sample:</u> LCS recoveries within laboratory limits <u>Soil Sample:</u> LCS recoveries within laboratory limits	1. Investigate the source of problem and document. 2. Re-analyze all samples processed with a LCS that is out of control.

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.
^b within 3 sigma of laboratory control charts. The laboratory should submit the control charts.

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Toxicity Characteristic Leaching Procedure (TCLP) for VOCs, SVOCs, Chlorinated Pesticides and Herbicides, and Metals by SW-846 Method 1311 and Analysis

Table of Contents	Pages
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SW-846 Methods 1311 TCLP and 8270C for Semivolatile Organic Compounds (SVOCs)	6 to 9
SW-846 Methods 1311 TCLP and 8081 for Chlorinated Pesticides	10 to 13
SW-846 Methods 1311 TCLP and 8151A for Chlorinated Herbicides	14 to 16
SW-846 Methods 1311 TCLP, 6010 for Metals, and 7470/7471 for Mercury	17 to 21

Table 1A. Summary of Holding Times and Preservation for TCLP Volatile Organic Compounds (VOCs) By SW-846 Method 1311

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation ^b
Volatile Organic Compounds (VOCs) in Water	<u>Technical for TCLP Extraction:</u> 14 days from collection; <u>Contract for TCLP Extraction:</u> 10 days from receipt at laboratory; <u>Technical and Contract Analysis:</u> 14 days from date of TCLP extraction to analysis.	Cool to 4°C ±2°C
VOCs in Soil ^{c, d}	<u>Technical and Contract for TCLP Extraction:</u> 48 hours cumulative from collection; (7 days if frozen) ^e <u>Technical and Contract of TCLP Extract:</u> 7 days from date of TCLP extraction to analysis.	Cool to 4°C ±2°C; sealed zero headspace containers. ^c

^a Individual target compounds are listed in Table 1B.

^b Preservatives should not be added to samples before extraction.

^c Freezing of soil samples requires contract approval.

^d Freezing of En Core™ samplers requires contract approval.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate the response factor (RF) and the concentration of individual analytes according to the equations specified in Sections 7.3 and 7.5 of SW-846 Method 8260B, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), (Revision 2). Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B. Target Compound List, Contract Required Quantitation Limits

*(CRQLs), and Regulatory and Spiking Levels for TCLP VOCs by GC/MS
Method 8260B*

<u>Analyte</u>	<u>CRQL $\mu\text{g/L}$</u>	<u>Regulatory Level mg/L</u>	<u>Spiking Level $\mu\text{g/L}$</u>
Benzene	10	0.5	50
Carbon tetrachloride	10	0.5	50
Chlorobenzene	10	100	50
Chloroform	10	6.0	50
1,4-Dichlorobenzene	10	7.5	50
1,2-Dichloroethane	10	0.5	50
1,1-Dichloroethene	10	0.7	50
Methyl ethyl ketone	10	200	50
Tetrachloroethene	10	0.7	50
Trichloroethene	10	0.5	50
Vinyl chloride	10	0.2	50

Table 2. Summary of Calibration Procedures for TCLP VOCs by SW-846 Method 8260B

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with 4-bromofluorobenzene (BFB)	Beginning of each 12 hour period during which standards and samples are analyzed	Ion abundance criteria in Table 4 of SW-846 Method 8260B	1. Identify the problem. 2. MS tune criteria must be met before calibration
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for RFs #20;	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV)	Following ICV, every 12-hour, and end of run	%D between RF of CCV and avg RFs from ICAL #±15%	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
System Performance Check Compound (SPCC)	With ICAL or CCV	RF for chloromethane, 1,1-dichloroethane, bromoform, \$0.10; chlorobenzene, 1,1,2,2-tetrachloroethane, \$0.30	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Calibration Check Compounds (CCC)	With ICAL or CCV	RSD for RFs #30%	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Internal Standards	Every standard, sample, blank, and QC sample	IS area within a factor of two of the IS area in the associated CCV (-50% to +100%)	1. Investigate the system; 2. Re-analyze all samples analyzed during a system malfunction
Retention time evaluation of CCV standards	Each analysis of CCV standard	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

^a The ICAL low standard must be above but near the CROL. The low ICAL standard must have a signal to noise ratio \$5:1. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package.

^b ICAL and CCV standards must contain all target analytes listed in Table 1B.

^c Report the retention time (RT) window for each analyte. Determine RT windows as ±3 x the standard deviation (SD) of the average initial calibration RT for each analyte.

Table 3. Summary of Internal Quality Control Procedures for TCLP VOCs BY SW-846 Method 8260B

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG ^a or one per batch of extraction fluid	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Reextract and/or reanalyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD) ^b	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	Sample extract: 75-125% of expected value; #25% RPD between MS and MSD	1. Report in case narrative
Surrogate Spikes ^{b, c}	Every sample, QC sample, standard and method blank	Sample extract: 85-115% of expected value, except for 1,2-dichloroethane (75-115%)	1. Reanalyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	Sample extract: 70-130% of expected value	1. Investigate the source of problem and document. 2. Reanalyze all samples processed with a LCS that is out of control.

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Spiking solution to be added after the TCLP extraction, immediately preceding analysis.

^c Toluene-d₈, BFB, 1,2-dichloroethane-d₄, and dibromofluoromethane

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Table 4A. Summary of Holding Times and Preservation for TCLP SVOCs by SW-846 Method 1311

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation ^b
Semivolatile Organic Compounds (SVOCs)	<u>Technical for TCLP Extraction:</u> 14 days from collection; <u>Contract for TCLP Extraction:</u> 10 days from receipt at laboratory; <u>Technical and Contract of TCLP Extract:</u> 7 days from date of TCLP extraction to preparative extraction; <u>Technical for Analysis:</u> 40 days from preparative extraction; <u>Contract for Analysis:</u> 35 days from preparative extraction.	Cool to 4°C ±2°C

^a Individual target compounds are listed in Table 4B.

^b Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Analyze the extract for SVOCs by SW-846 Method 8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), (Revision 3.0). Calculate the concentration of individual analytes according to Section 7.7.2 of EPA Method 8270C. Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 4B. *Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP SVOCs by SW-846 Method 8270C*

<u>Analyte</u>	<u>CRQL µg/L</u>	<u>Regulatory Level mg/L</u>	<u>Spiking Level µg/L</u>
o-Cresol (2-Methylphenol)	10	200	50
m-Cresol (3-Methylphenol)	10	200	50
p-Cresol (4-Methylphenol)	10	200	50
2,4-Dinitrotoluene	10	0.13	50
Hexachlorobenzene	10	0.13	50
Hexachloro-1,3-butadiene	10	0.5	50
Hexachloroethane	10	3.0	50
Nitrobenzene	10	2.0	50
Pentachlorophenol	25	100	75
Pyridine	25	5.0	75
2,4,5-Trichlorophenol	25	400	75
2,4,6-Trichlorophenol	10	2.0	50

Table 5. *Summary of Calibration Procedures for TCLP SVOCs by SW-846 Method*

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with decafluorotriphenylphosphine (DFTPP)	Beginning of each 12-hour period during which standards and samples are analyzed	Ion abundance criteria in Table 3 of Method 8270C, Revision 3.0	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) <i>a, b, c</i>	Initially; whenever required, due to failure of CCV	%RSD for RRFs #30%; or correlation coefficient (<i>r</i>) generated by the linear regression must be ≤ 0.99 for all analytes	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV) ^d	Beginning of every 12-hour period, and end of run	%D between RRF of CCV and avg RRFs from ICAL #30%; or $\pm 30\%$ of true value for linear regression	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Integrated areas of Internal Standards (IS)	Each analysis	Area must be within -50 to 100 percent. Retention time ± 0.33 minutes of CCV IS retention times.	1. Re-analyze samples with internal standard -50 percent and greater than 100 percent
Retention time evaluation of all standard, surrogate, and sample analytes	Each analysis	± 3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples out of control limits

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b ICAL and continuing CAL standards must contain all target analytes listed in Table 4B.

^c Report the retention time window for each analyte. Determine retention time windows as ± 3 x the standard deviation of the average initial calibration retention time for each analyte.

^d If some compounds are beyond the control limits of the CCV and these target compounds are detected in samples and 10 percent or less of these analytes are beyond control limits, a single point calibration may be used to quantify the out-of-control analytes.

Table 6. Summary of Internal Quality Control Procedures for TCLP SVOCs by SW-846 Method 8270C

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG ^a or one per batch of extraction fluid	< CRQL for each compound	1. Investigate the source of contamination and document. 2. Re-analyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	MS and MSD recoveries (65-135%) and RPD 30%	1. Report in case narrative
Surrogate Spikes ^b	Every sample, standard and method blank	Surrogate recoveries within laboratory limits	1. Re-analyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS) ^b	One per SDG	LCS recoveries within laboratory limits	1. Investigate the source of problem and document. 2. Re-analyze all samples processed with a LCS that is out of control.

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Within 3 sigma of laboratory control charts. The laboratory should submit the control charts.

Dilute and re-analyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Table 7A. Summary of Holding Times and Preservation for TCLP Chlorinated Pesticides by SW-846 Method 1311

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation ^b
Chlorinated Pesticides	<p><u>Technical for TCLP Extraction:</u> 14 days from collection;</p> <p><u>Contract for TCLP Extraction:</u> 10 days from receipt at laboratory;</p> <p><u>Technical and Contract of TCLP Extract:</u> 7 days from date of TCLP extraction to preparative extraction;</p> <p><u>Technical for Analysis:</u> 40 days from preparative extraction;</p> <p><u>Contract for Analysis:</u> 35 days from preparative extraction.</p>	Cool to 4°C ±2°C

^a Individual target compounds are listed in Table 7B.

^b Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate the calibration factors (CF) of single component pesticides according to Section 7.4 of SW-846 Method 8081, Organochlorine Pesticides and PCBs as Aroclors by Gas Chromatography: Capillary Column Technique, (Revision 0). Calculate sample results using the analyte CFs from the midpoint standard of the associated initial calibration curve. Perform sample quantitation for multiple components pesticides according to Section 7.6 of SW-846 Method 8081. Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 7B: Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP Chlorinated Pesticides by

SW-846 Method 8081

<u>Analyte</u>	<u>CROL $\mu\text{g/L}$</u>	<u>Regulatory Level mg/L</u>	<u>Spiking Level $\mu\text{g/L}$</u>
Chlordane (Technical)	0.5	0.03	5
Endrin	0.1	0.02	1
Heptachlor	0.05	0.008	0.5
Heptachlor epoxide	0.05	0.008	0.5
Lindane (gamma-BHC)	0.05	0.4	0.5
Methoxychlor	0.5	10	5
Toxaphene	5	0.5	50

Table 8. Summary of Calibration Procedures for TCDF Organochlorine Pesticides and Polychlorinated Biphenyls (PCBs) by SW-846 Method 8081

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 3 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for CFs #20% (#30% for Surrogate compounds)	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL	Beginning of each day, after every 10 samples, and end of run	% D between CF of CCV and avg CFs from ICAL #25%	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Endrin and 4,4'-DDT Breakdown	Beginning and end of analytical sequence	#20% each analyte or #30% combined analytes	1. Investigate source of the problem and document 2. If either Endrin, 4,4'-DDT, or their breakdown products were detected, re-analyze the samples

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio \$5:1. If this requirement cannot be met, the laboratory must submit a method detection limit (MDL) study as part of the data package.

^b For ICAL, prepare individual standard mixtures A and B (IND A and IND B) containing the single component pesticides specified in Table 9 of SW-846 Method 8081 at three concentration levels. For multiple response pesticides, including toxaphene and Aroclors (except 1016 and 1260), prepare separate ICAL standards at the following concentration levels: Aroclors (except 1221) at 100 ng/mL; Aroclor-1221 at 200 ng/mL; and toxaphene at 500 ng/mL. Aroclor-1016 and Aroclor-1260 may be combined into a single standard solution. Spike all calibration standards with the surrogate compounds discussed in the following Table 9 at a concentration of 20 ng/mL.

^c Report the retention time (RT) window for each analyte. For multiple component pesticides, calculate the RT window for 5 major peaks from the initial calibration standard analysis. Determine RT windows for both single and multiple component pesticides using the following guidelines:

Column Type	RT Window in Minutes
Packed Column	#± 2%
Mega bore or wide bore capillary column	±0.05 for tetrachloro-m-xylene through Aldrin ±0.07 for compounds which elute after Aldrin ±0.1 for decachlorobiphenyl

Table 9. Summary of Internal Quality Control Procedures for TCDF Organochlorine Pesticides and Polychlorinated

Biphenyls (PCBs) by SW-846 Method 8081

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum) or one per batch of extraction fluids.	< CRQL for each compound	1. Investigate source of contamination and document 2. Re-extract and re-analyze all samples processed with a non-compliant method blank
Surrogate ^b	Every standard, sample, method blank and QC sample at 10 times CRQL	60-150% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries
Matrix Spike and Matrix Spike Duplicate (MS/MSD) ^c	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	65-135% of expected value; #30 RPD between MS and MSD	1. Address in narrative

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Spike each standard, sample, and blank with 1mL of a solution containing 0.2 µg/mL each of tetrachloro-m-xylene and decachlorobiphenyl

^c Spike MS/MSD samples with 1mL of a solution containing the following compounds and levels:

Target compound	Concentration (µg/mL)	Target Compound	Concentration (µg/mL)
(-BHC	0.5	Heptachlor	0.5
4,4'-DDT	1.0	Aldrin	0.5
Endrin	1.0	Dieldrin	1.0

Dilute and re-analyze samples with one or more analytes at concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Perform confirmation analyses on a column of a phase different from that used for quantitation. Confirmation analyses must meet all instrument calibration criteria and blank acceptance criteria specified in Table 8, above.

Table 10A. Summary of Holding Times and Preservation for TCLP Chlorinated Herbicides by SW-846 Method 1311

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation ^b
Chlorinated Herbicides	<u>Technical for TCLP Extraction:</u> 14 days from collection; <u>Contract for TCLP Extraction:</u> 10 days from receipt at laboratory; <u>Technical and Contract of TCLP Extract:</u> 7 days from date of TCLP extraction to preparative extraction; <u>Technical for Analysis:</u> 40 days from preparative extraction; <u>Contract for Analysis:</u> 35 days from preparative extraction.	Cool to 4°C ±2°C

^a Individual target compounds are listed in Table 10B.

^b Preservatives should not be added to samples before extraction.

Data Calculations and Reporting Units:

Determine the percent solid as specified in Section 7.1 of SW-846 Method 1311 and report the result as percent solid. Extract the second sample/aliquot according to Section 7.3 of SW-846 Method 1311.

Calculate calibration factors and sample results according to Sections 7.7 and 7.8 of SW-846 Method 8151B, Chlorinated Herbicides by Gas Chromatography, (Revision 1). Report final analyte concentration in units of micrograms per liter (µg/L). Report results that are less than 10 µg/L to 1 significant figure, and results that are greater than or equal to 10 µg/L to 2 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 10B. Target Compound List, Contract Required Quantitation Limits (CRQLs), and Regulatory and Spiking Levels for TCLP Chlorinated Herbicides by SW-846 Method 8150B

Analyte	CRQL µg/L	Regulatory Level mg/L	Spiking Level µg/L
2,4-D	12	10	60-100
2,4,5-TP (Silvex)	7	10	35-100

Table 11. Summary of Calibration Procedures for TCLP Chlorinated Herbicides by

SW-846 Method 8151

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) a, b, c	Initially; whenever required, due to failure of CCV	RSD for CFs #20%; or, if using a linear calibration curve, a correlation coefficient (r) of ≥ 0.99 for each compound	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Continuing Calibration Verification (CCV) at midpoint of ICAL (Separate source from ICAL standards)	Beginning of each 12-hour time period, after every 10 samples and end of run	%D between calculated and nominal amount for each compound must be # $\pm 5.0\%$	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV
Retention time evaluation for CCV standards	Each analysis of CCV standards	± 3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio $\geq 5:1$. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b Report the retention time window for each analyte. Determine retention time windows as ± 3 x the standard deviation (SD) of the average initial calibration retention time for each analyte.

^c ICAL and continuing CAL standards must contain all surrogate compounds and target analytes listed in Table 10B.

Table 12. Summary of Internal Quality Control Procedures for TCIP Chlorinated Herbicides by SW-846 Method 8151

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum) per analytical instrument	< CRQL for each compound	1. Investigate source of contamination and document 2. All samples processed with a method blank that is out of control must be re-extracted and re-analyzed
Surrogate Spike	Every standard, sample and method blank at 10 times CRQL	75-125% of expected value	1. Re-analyze all samples with non-compliant surrogate recoveries 2. If re-analysis does not solve the problem, re-extract and re-analyze
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum) containing all analytes chosen from Table 10B	40-160% of expected value for dinoseb and 65-135% of expected value for other target analytes; #30 RPD between MS and MSD	1. Report in Case Narrative
Laboratory Control Sample (LCS)	One LCS per batch or SDG	40-160% for dinoseb; 80-120% for other target analytes	1. Re-extract and re-analyze all samples processed with an out-of-control LCS

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.

Second column confirmation is required for all positive results. Confirmation must be performed on a column of a phase different from that used for quantitation. Confirmation analyses must meet all calibration criteria specified in Table 2 and blank acceptance criteria specified in Table 3 of the SW-846 Method 8151.

Table 13A. Summary of Holding Times and Preservation for TCLIP Metals by SW-846 Method 1311

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Metals (except mercury)	<u>Technical</u> : 180 days from date of collection to TCLP extraction and another 180 days from date of TCLP extraction to analysis; <u>Contract</u> : TCLP extraction 26 days from sample receipt at laboratory and analysis within 26 days of extraction	Cool to 4°C ±2°C After extraction and filtration, pH <2 with nitric acid
Mercury	<u>Technical</u> : 28 days from date of collection to TCLP extraction and another 28 days from date of TCLP extraction to analysis; <u>Contract</u> : TCLP extraction 26 days from sample receipt at laboratory and analysis within 26 days of extraction	Cool to 4°C ±2°C After extraction and filtration, pH <2 with nitric acid

^a Individual target compounds are listed in Table 13B.

Data Calculations and Reporting Units:

Calculate the sample results according to the protocols of the appropriate analytical method: SW-846 Method 6010B (ICP) Section 7.6, SW-846 Methods 7470/7471 (CVAA) Sections 7.5 and 7.6, respectively, and SW-846 Method 1311 (TCLP Extraction) Section 7.2.14.

Report sample results in concentration units of milligrams per liter (mg/L).

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

TABLE 13B. *Target Compound List, CAS Numbers, Contract Required Detection Limits, Regulatory and Spiking Levels for TCLP Metals by SW-846 Method 6010 and SW-846 Method 7470/7471*

COMPOUND	CAS No.	CRDL (mg/L)	Regulatory Level (mg/L)	Spiking Level (mg/L)
Arsenic	7440-38-2	0.50	5.0	2.5 - 5.0
Barium	7440-39-3	1.0	100	50 - 100
Cadmium	7440-43-9	0.10	1.0	0.5 - 1.0
Chromium	7440-47-3	0.50	5.0	2.5 - 5.0
Lead	7439-92-1	0.50	5.0	2.5 - 5.0
Mercury	7439-97-6	0.02 ^a	0.2	0.1 - 0.2
Selenium	7782-49-2	0.10	1.0	0.5 - 1.0
Silver	7440-22-4	0.50	5.0	2.5 - 5.0

^a Mercury analysis is to be performed using 10 mL aliquots diluted to 100 mL.
The CRDL has been adjusted to account for this 10 X dilution.

Table 14A. Summary of Calibration Procedures for TCLP Metals by SW-846 Method 6010

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
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Initial Calibration (minimum blank + 1 calibration standard) (ICAL)	Initially, Daily; whenever required, due to failure of CCV	Acceptable ICV, CRDL, and ICB standards	1. Terminate analysis 2. Re-calibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	±10% from expected concentration	1. Terminate analysis and identify and document problem 2. Reprep and re-analyze ICV and all associated samples 3. Re-calibrate and re-analyze repressed ICV and all associated samples
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine Source of contamination 3. Reprep ICB and CCB 4. Re-analyze all samples associated with a contaminated blank
Continuing Calibration Verification (CCV)	Before samples, after every 10 samples, and end of run	± 10% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last acceptable CCV
Contract Required Detection Limit Verification Standard (CRI)	After ICV and before sample analysis	±35% from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back to last compliant CCV
ICP Interference Check Sample (ICS)	Run at start and finish of daily run or twice per 8 hours	± 20% from true value concentration	1. Reprep and re-analyze standard 2. Re-calibrate, verify and re-analyze all associated samples

Table 14B. Summary of Calibration Procedures for TCLP Mercury by SW-846 Method
7470/7471

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 standards) (ICAL) ^a	Initially, each analytical batch; whenever required, due to failure of CCV	r \leq 0.995	1. Terminate analysis 2. Re-calibrate and verify b analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Different source from ICAL standards)	Daily, immediately following ICAL and prior to sample analysis	$\pm 20\%$ from expected concentration	1. Terminate analysis and id document problem 2. Re-prepare and re-analyze ICV associated samples 3. Re-calibrate and re-analyze ICV and all associated sa
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Determine source of conta 3. Re-prepare ICB and CCB 4. Re-analyze all samples as with a contaminated blank
Continuing Calibration Verification (CCV)	Before Samples, after every 10 samples, and end of run	$\pm 20\%$ from expected concentration	1. Re-calibrate and verify 2. Re-analyze samples back t acceptable CCV
Contract Required Detection Limit Verification Standard (CRA)	After ICV, and before sample analysis	$\pm 35\%$ from expected concentration	1. Re-prepare and re-analyze sta 2. Re-calibrate and verify

^a The ICAL low standard must be at the CRDL.

Table 15. Summary of Internal Quality Control Procedures for TCLP Metals Analysis by SW-846 Method 6010 and SW-846 Method 7470/7471

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	One per SDG ^a or per batch of extraction fluid ^b	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all affected samples must be prepared again with another method blank and re-analyzed
Duplicate Sample (DUP)	One per batch or SDG ^{a, b}	RED <± 20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike Sample (MS)	One per batch or SDG ^{a, b, c}	± 25% from expected value ^d	1. A post-digestion spike must be performed for analytes that exceed limits.
Laboratory Control Sample (LCS) ^e	One per SDG ^a or per batch of extraction fluid ^b	± 20% from expected concentration	1. Terminate analysis and identify and document the problem 2. Re-analyze all associated samples
Serial Dilution Sample (5 X Dilution) (ICP only)	One per batch or SDG ^{a, b}	± 10% difference from original results for analytes greater than 50 X IDL	1. Flag associated data with a "B"

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Minimum requirement is the analysis of 1 QC sample per 20 samples.

^c Spiking solution must contain all analytes within the spiking ranges listed in Table 13B. Matrix spikes

are to be added after filtration of the TCLP extract and before acidification.

^d An exception to this rule is granted in situations where the sample concentration exceeds the spike

concentration by a factor of 4.

^e LCS spike solution must be from a different source than the calibration standards.

Dilute and re-analyze samples with concentrations exceeding the range of the calibration curve. Results for such

re-analyses should fall within the mid-range of the calibration curve. Report results and submit documentation

for both analyses.

VOLATILE ORGANIC COMPOUNDS (VOCs)

SW-846 Method 8260

Table 1A. Summary of Holding Times and Preservation for Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry

Analytical Parameter ^a	Technical and Contract Holding Times	Preservation
Volatile Organic Compounds (VOCs) in Water	<u>Technical</u> : 7 days from collection; <u>Contract</u> : 5 days from receipt at laboratory	Cool to 4EC ±2EC;
VOCs in Water	<u>Technical</u> : 14 days from collection; <u>Contract</u> : 10 days from receipt at laboratory	HCl to pH <2; Cool to 4EC ±2EC
VOCs in Soil	<u>Technical</u> : 48 hours <u>Contract</u> : 48 hours	Cool to 4EC ±2EC; sealed zero headspace containers; freezing can extend the holding time ^b
VOCs in Soil	<u>Technical</u> : 14 days from collection; <u>Contract</u> : 10 days from receipt at laboratory	Preserved samples: in methanol ^c or sodium bisulfate ^d

^a Individual target compounds are listed in Table 1B.

^b Freezing the sample can extend the holding time; however, 48 hours unfrozen holding time will be considered cumulative.

^c Use Method 5030 for purge and trap.

^d Use Method 5035 for purge and trap.

Data Calculations and Reporting Units:

Calculate the response factor (RF) and the concentration of individual analytes according to the equations specified in Sections 7.3.4 of Method 8260. Report water sample results in concentration units of micrograms per liter (Fg/L).

Report soil sample results on a dry-weight basis in micrograms per kilogram (Fg/kg). Report percent solid and percent moisture to the nearest whole percentage point.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;
- b) If the number following those to be retained is greater than 5, round up; or
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 1B. Target Compound List, CAS Numbers, and Contract Required Quantitation Limits for Volatile Organic Compounds by Method 8260

Analyte	CAS Number	CROL Fg/L ^a	CROL Fg/Kg ^b
Benzene	71-43-2	1	5
Bromobenzene	108-86-1	1	5
Bromochloromethane	74-97-5	1	5
Bromodichloromethane	75-27-4	1	5
Bromoform	75-25-2	1	5
Bromomethane	74-83-9	1	5
n-Butylbenzene	104-51-8	1	5
sec-Butylbenzene	135-98-8	1	5
tert-Butylbenzene	98-06-6	1	5
Carbon tetrachloride	56-23-5	1	5
Chlorobenzene	108-90-7	1	5
Chlorodibromomethane	124-48-1	1	5
Chloroethane	75-00-3	1	5
Chloroform	67-66-3	1	5
Chloromethane	74-87-3	1	5
2-Chlorotoluene	95-49-8	1	5
4-Chlorotoluene	106-43-4	1	5
1,2-Dibromo-3-chloropropane	96-12-8	1	5
1,2-Dibromoethane	106-93-4	1	5
Dibromomethane	74-95-3	1	5
1,2-Dichlorobenzene	95-50-1	1	5
1,3-Dichlorobenzene	541-73-1	1	5
1,4-Dichlorobenzene	106-46-7	1	5
Dichlorodifluoromethane	75-71-8	1	5
1,1-Dichloroethane	75-34-3	1	5
1,2-Dichloroethane	107-06-2	1	5
1,1-Dichloroethene	75-35-4	1	5
cis-1,2-Dichloroethene	156-59-2	1	5
trans-1,2-Dichloroethene	156-60-5	1	5

1,2-Dichloropropane	78-87-5	1	5
2,2-Dichloropropane	594-20-7	1	5
1,3-Dichloropropane	142-28-9	1	5
1,1-Dichloropropene	563-58-6	1	5
Ethylbenzene	100-41-4	1	5
Hexachlorobutadiene	87-68-3	1	5
Isopropylbenzene	98-82-8	1	5
p-Isopropyltoluene	99-87-8	1	5
Methylene chloride	75-09-2	1	5
Naphthalene	91-20-3	1	5
n-Propylbenzene	103-65-1	1	5
Styrene	100-42-5	1	5
1,1,1,2-Tetrachloroethane	630-20-6	1	5
1,1,2,2-Tetrachloroethane	79-34-5	1	5
Tetrachloroethene	127-18-4	1	5
Toluene	108-88-3	1	5
1,2,4-Trichlorobenzene	120-82-1	1	5
1,2,3-Trichlorobenzene	87-61-6	1	5
1,1,1-Trichloroethane	71-55-6	1	5
1,1,2-Trichloroethane	79-00-5	1	5
Trichloroethene	79-01-6	1	5
Trichlorofluoromethane	75-69-4	1	5
1,2,3-Trichloropropane	96-18-4	1	5
1,2,4-Trimethylbenzene	95-63-6	1	5
1,3,5-Trimethylbenzene	108-67-8	1	5
Vinyl chloride	75-01-4	1	5
o-Xylene	95-47-6	1	5
m-Xylene	108-38-3	1	5
p-Xylene	106-42-3	1	5
Methyl-t-butyl ether	163-40-44	1	5
Dichlorofluoromethane	75-43-4	1	5

^a Based on 25 mL water purge. ^b Based on wet weight

Table 2. Summary of Calibration Procedures for VOCs by SW-846 Method 8260

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
GC/MS Tuning with BFB	Beginning of each 12 hour period during which standards samples are analyzed	Ion abundance criteria in Table 4 of Method 8260	1. Identify the problem. 2. MS tune criteria must be met before any calibration standards, samples, blanks, or QC samples are analyzed
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) ^{a, b, c}	Initially; whenever required, due to failure of CCV	RSD for REs #20%;	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Continuing Calibration Verification (CCV)	Following ICV, every 12-hour, and end of run	%D between RF of CCV and avg RFs from ICAL #15%	1. Recalibrate and verify 2. Reanalyze samples back to last good CCV
System Performance Check Compound (SPCC)	With ICAL or CCV	RF for chloromethane, 1,1-dichloroethane, bromoform, \$0.10; chlorobenzene, 1,1,2,2-tetrachloroethane, \$0.30	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Calibration Check Compounds (CCC)	With ICAL or CCV	RSD for RFs #30%	1. Terminate analysis 2. Recalibrate and verify before sample analysis
Internal Standards	Each analysis of CCV	-50 to +100%	1. Re-analyze all samples analyzed while system was out-of-control
Retention time evaluation of CCV standards	Each analysis of CCV standard	±3 x the SD of the avg ICAL RT for each analyte	1. Re-calibrate and verify 2. Re-analyze samples back to last good CCV

^a The ICAL low standard must be above but near the CRQL. The low ICAL standard must have a signal to noise ratio 5:1. If this requirement cannot be met, the laboratory must submit a MDL study as part of the data package.

^b ICAL and continuing CAL standards must contain all target analytes listed in Table 1B.

^c Report the retention time window for each analyte. Determine retention time windows as ±3 x the standard deviation of the average initial calibration retention time for each analyte.

Table 3. Summary of Internal Quality Control Procedures for VOCs by SW-846 Method 8260

QC Element	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	Each 12-hour time period, minimum of one per SDG ^a	< CROL for each compound	<ol style="list-style-type: none"> Investigate the source of contamination and document. Reanalyze all samples processed with a blank that is out of control.
Matrix Spike and Matrix Spike Duplicate (MS/MSD)	One MS/MSD set per batch or SDG (1 MS/MSD set per 20 samples minimum)	<p>Water Sample: 65-135% of expected value; #30% RPD between MS and MSD</p> <p>Soil Sample: 50-150% of expected value; #50% RPD between MS and MSD</p>	<ol style="list-style-type: none"> Report in case narrative
Surrogate Spikes: ^b	Every sample, standard and method blank	<p>Water Sample: 85-115% except for 1,2-dichloroethane (75-115%) of expected value</p> <p>Soil Sample: 70-125% of expected value</p>	<ol style="list-style-type: none"> Reanalyze all samples with non-compliant surrogate recoveries
Laboratory Control Sample (LCS)	One per SDG	<p>Water Sample: 70-130% of expected value</p> <p>Soil Sample: 65-135% of expected value</p>	<ol style="list-style-type: none"> Investigate the source of problem and document. Reanalyze all samples processed with a LCS that is out of control.

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

^b Toluene-d₈, BFB, 1,2-dichloroethane-d₄, and Dibromofluoromethane

Dilute and reanalyze samples which contain one or more target analytes at concentrations above the initial calibration range. Results for such reanalyses should fall within the mid-range of the calibration curve.

Report results and submit documentation for both analyses.

**Appendix B -
Sample Chain-of-Custody Form**



CHAIN OF CUSTODY

PAGE _____ OF _____

WESTBORO, MA
TEL: 508-898-9220
FAX: 508-898-9193

MANSFIELD, MA
TEL: 508-822-9300
FAX: 508-822-3288

Client Information

Client:

Address:

Phone:

Fax:

Email:

These samples have been previously analyzed by Alpha

Other Project Specific Requirements/Comments/Detection Limits:

Project Information

Project Name:

Project Location:

Project #:

Project Manager:

ALPHA Quote #:

Turn-Around Time

Standard RUSH (only confirmed if pre-approved)

Date Due: _____ Time: _____

Date Rec'd in Lab:

ALPHA Job #:

Report Information - Data Deliverables

FAX EMAIL

ADEX Add'l Deliverables

Same as Client info PO #:

Regulatory Requirements/Report Limits

State / Fed Program _____

Criteria _____

ANALYSIS

TOTAL # BOTTLES

SAMPLE HANDLING

- Filtration _____
 - Done
 - Not needed
 - Lab to do
 - Preservation
 - Lab to do
- (Please specify below)

Sample Specific Comments

Container Type

Preservative

Date/Time

Relinquished By:

Received By:

Date/Time

Please print clearly, legibly and completely. Samples can not be logged in and turnaround time clock will not start until any ambiguities are resolved. All samples submitted are subject to Alpha's Terms and Conditions. See reverse side.

Instructions

Where to send the report.
Enter phone, fax and
email to info here

Enter Project Information and
especially Alpha Quote #

Indicate additional report
requirements other than
standard mail.

Indicate where bill is to be
sent and include PO
number.

Indicate if Standard or Rush Request. Indicate
the Date and Time Due!

Indicate Sample ID for
each sample, date and
time collected, matrix
type, sampler and
check off for each
analysis requested.

- Matrix/Source Codes:
- I= Influent
 - E= Effluent
 - DW = Drinking Water
 - GW = Ground Water
 - SW = Surface Water
 - MW = Monitoring Well
 - RO= Run-off
 - L= Lake/Pond/River
 - B= Bottom Sediment
 - S= Soil
 - SG= Sludge
 - O= Oil
 - W= Wipe
 - SE= Sediment
 - T= Tissue
 - X1(Other)

Enter Special Instructions such as
specific Detection Limits here!

List Analyses Requested. Be specific
Example: 8260 LOW
EPH Deluxe

Indicate if Filtration/Preservation is done
or is needed and list in comment section
below for each sample.

Enter Container Type and Preservative Code

- Container Code
- P= Plastic
 - A= Amber glass
 - V= Vial
 - G= Glass
 - B= Bacteria cup
 - C= Cube
 - O= Other
 - E= Encone
 - D= BOD Bottle
- Preservative Code
- A= None
 - B= HCl
 - C= HNO₃
 - D= H₂SO₄
 - E= NaOH
 - F= MeOH
 - G= NaHSO₄
 - H= Na₂S₂O₃
 - I= Ascorbic Acid
 - J= NH₄Cl
 - K= Zn Acetate
 - L= NH₄Cl Phosphate
 - O= Other

Signatures, Date & Time when
relinquishing or receiving.

Terms & Conditions:

In the absence of a written agreement to the contrary, this order constitutes an acceptance by the Client of Alpha Analytical, Inc. (ALPHA)'s offer to do business under these Terms and Conditions, and agrees to be bound by these conditions. Any terms and conditions from Client's that do not conform to the terms and conditions contained herein shall be deemed invalid and unenforceable, unless accepted in writing by ALPHA. This order shall not be valid unless it contains sufficient specifications to enable ALPHA to carry out the Client's requirements. Samples must be accompanied by: a) adequate instruction as to the quantity and type of analysis requested, and b) reporting and billing address information. Upon timely delivery of samples, ALPHA will use its best efforts to meet mutually agreed turnaround times, calculated from the point in time when ALPHA has determined that it can proceed with the defined work to be done (Sample Delivery Acceptance). ALPHA reserves the right, to refuse or revoke Sample Delivery Acceptance for any sample which in the sole judgment of ALPHA: a) is unsuitable volume; b) may pose a risk or become unsuitable for handling, transport or processing for any health, safety, environmental or any other reason; c) holding times cannot be met.

Client agrees to pay for all applicable charges to process this order. Payment in advance is required for all Clients except those whose credit has been established with ALPHA. For Clients with approved credit, payment terms are Net 30 days from the date of the invoice by ALPHA. All overdue payments are subject to an interest and service charge of one and one half percent (1.5%) (Or the maximum rate permissible by law, whichever is lesser) per month or portion thereof from the due date until the date of payment. ALPHA may suspend work and withhold delivery of data until this order at any time in the event that the Client fails to make timely payment of its invoices. Client shall be responsible for all costs and expenses of collection including reasonable attorney's fees. Data or information provided to ALPHA or generated by services performed under this agreement shall only become the property of the Client upon receipt in full by ALPHA of payment for the entire Order.

In no event shall ALPHA have any responsibility or liability to the Client for any failure or delay in performance by ALPHA which results, directly or indirectly in whole or in part, from any cause or circumstance beyond the reasonable control of ALPHA.

ALPHA shall dispose of the Client's samples 30 days after the analytical report is issued, unless instructed to store them for an alternate period of time or return such samples to the Client. The return of samples will be at the Client's own expense.